

Plating experiments under high speed plating conditions (Jet Plating)

The electrodeposition bath according to the claimed invention had been especially developed for application under high speed plating conditions, so called Jet Plating.

- 5 In Jet Plating, the electrolyte is shot by a jet in the form of a coherent stream onto the substrate which acts as the cathode (see Figure 1). The jet forms the anode.

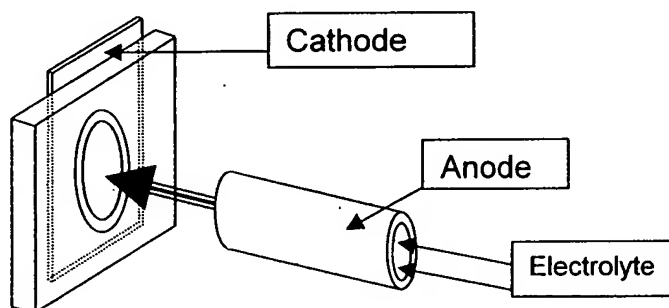


Figure 1

For the measurements reported below, we used a laboratory jet plating apparatus (Jet Lab). This jet plating arrangement is described in

- 10 Raub C. J.: Jet Plating-Laboratory Simulation & Control; Trans. Inst. Metal Fin., 68 (1990), S.115ff
Abstract: "Report on construction of a small pumped cell which simulates jet plating and of results obtained with the cell for various electrolytes. Limiting current densities are given and conditions for obtaining good deposits."
- 15 Jet plating offers the advantage to work with high current densities up to more than 100 A/dm² and allows a better discrimination between the various plating baths.
- In the following experiments plating speed and current efficiency were determined versus current density. The current density was increased from 10 A/dm² in steps of 10 A/dm². The basic electrolyte (BE) used was a standard gold bath containing 12 g/l Au.
- 20 The composition in detail is given below:
- 12 g/l of Au, as potassium gold(I)-cyanide
 - 0,5 g/l of Co, as cobalt sulfate
 - 180 g/l of citric acid
 - pH adjusted to 4,2 with potassium hydroxide

The working range was determined by inspection of the deposits. When the deposit at one current density showed so-called burnt marks then the upper limit of the working range was set to be equal to the current density of the previous step.

Results

5 1. Investigation of

- basic electrolyte (BE);
- BE + octyl sulfate;
- BE + pyridine-3- sulfonic acid
- BE + both additives

10 Figure 2 shows the series of test pieces resulting from the experiments. The upper row shows the test pieces for deposition from the basic electrolyte only. At 30 A/dm² a burnt ring can be observed which is not allowed. Thus the upper working range was set to 20 A/dm².

15 The second row shows the situation for deposition from the basic electrolyte plus the addition of 2 g/l of octyl sulfate. No increase of working range can be seen. Plating speed and current efficiency remain nearly the same.

20 The third row shows the results for deposition from the basic electrolyte plus the addition of 4 g/l pyridine-3-sulfonic acid. The striking point here is that the working range is increased to 70 A/dm². But it should be noted that plating speed and current efficiency e.g. at 20 A/dm² are reduced in comparison to the deposition from the basic electrolyte alone. This confirms that Biberbach is in error.

25 Plating speed can only be increased by increasing the current density. At a current density of 70 A/dm² the plating speed has increased to 7.6 µm/min but at the same time current efficiency has decreased to only 15,1 %. We get here only a net increase in plating speed because the higher current density overcompensates the adverse effect of the reduced current efficiency.

30 The fourth row shows the results for deposition from the basic electrolyte plus the combination of both additives according to one embodiment of the invention. The working range is further increased to 100 A/dm². At the same current density the current efficiency is higher than in the third row. This results in an increased plating speed.

These findings were not expected because the addition of octyl sulfate alone to the basic electrolyte hardly changes anything. But in combination with pyridine-3-sulfonic acid a remarkable improvement is achieved.

5 In Figure 3 the data given in Figure 2 are presented as a diagram of plating speed and current efficiency versus current density. From this presentation it can clearly be seen that the current efficiency drops with increasing current density.

2. Investigation of

- BE + pyridine-3-sulfonic acid
- BE + pyridine-3-sulfonic acid + 4 g/l nicotinic acid
- 10 • BE + 2 g/l octyl sulfate acid + 2 g/l hexyl sulfonate

These series of experiments were performed to investigate the results of combining two gloss additives from the first group of compounds (pyridine-3-sulfonic acid and nicotinic acid) and the results of combining two gloss additives according to formula I (octyl sulfate and hexyl sulfonate).

15 The results are given in the diagram of figure 4. For comparison also the curves for the basic electrolyte plus pyridine-3-sulfonic acid are given. It can be seen that the combination of pyridine-3-sulfonic acid with nicotinic acid lowers the current efficiency and hence the plating speed compared to pyridine-3-sulfonic acid alone. This is contrary to the combination of pyridine-3-sulfonic acid with octyl sulfate according to one embodiment of the invention.

20 The combination of octyl sulfate with hexyl sulfonate, both of which belong to compounds according to formula I, doesn't neither improve the working range nor current efficiency or plating speed.

25 This shows that it is important to make the right selection when combining two gloss additives in order to increase working range and plating speed.

EXHIBIT A

US Patent Application 10/009,467; PCT-Application EP00/03993; internal code: 990073 GV

Date 25 November 2003

Composition of the gold-cobalt electrolyte ("basic electrolyte"):

12 g/l of Au as potassium gold(I)-cyanide 0.5 g/l of Co as cobalt sulphate 180 g/l of citric acid
pH adjusted to 4.2 with potassium hydroxide

Operating conditions:

High-Speed ("jet") plating in a JetLab (600 l/h)
12 g/l Au 60 °C pH 4.2

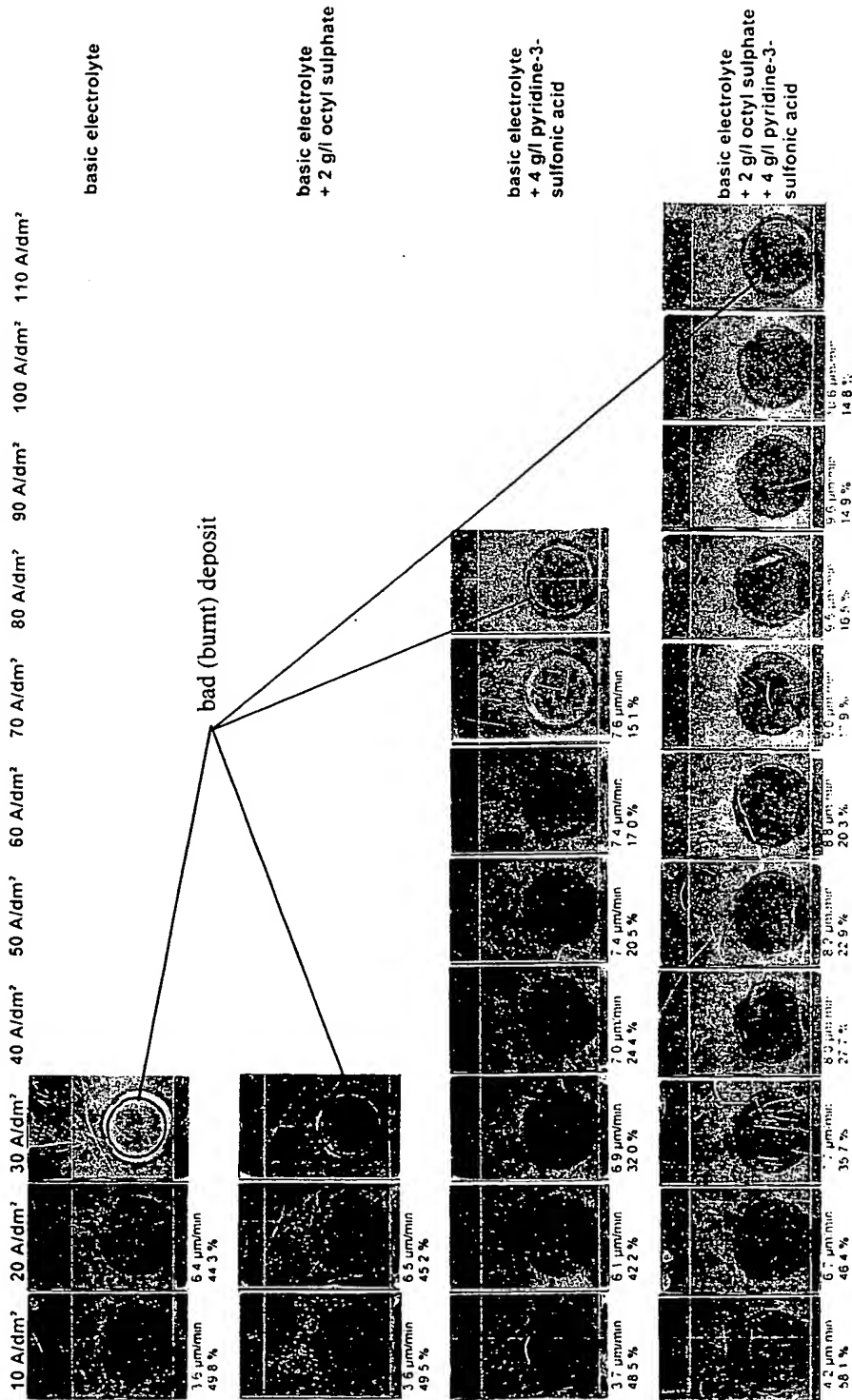


Figure 2

EXHIBIT A

AuCo Electrolyte (12 g/l Au; 60 °C; pH 4.2; JetLab 600 I/h)

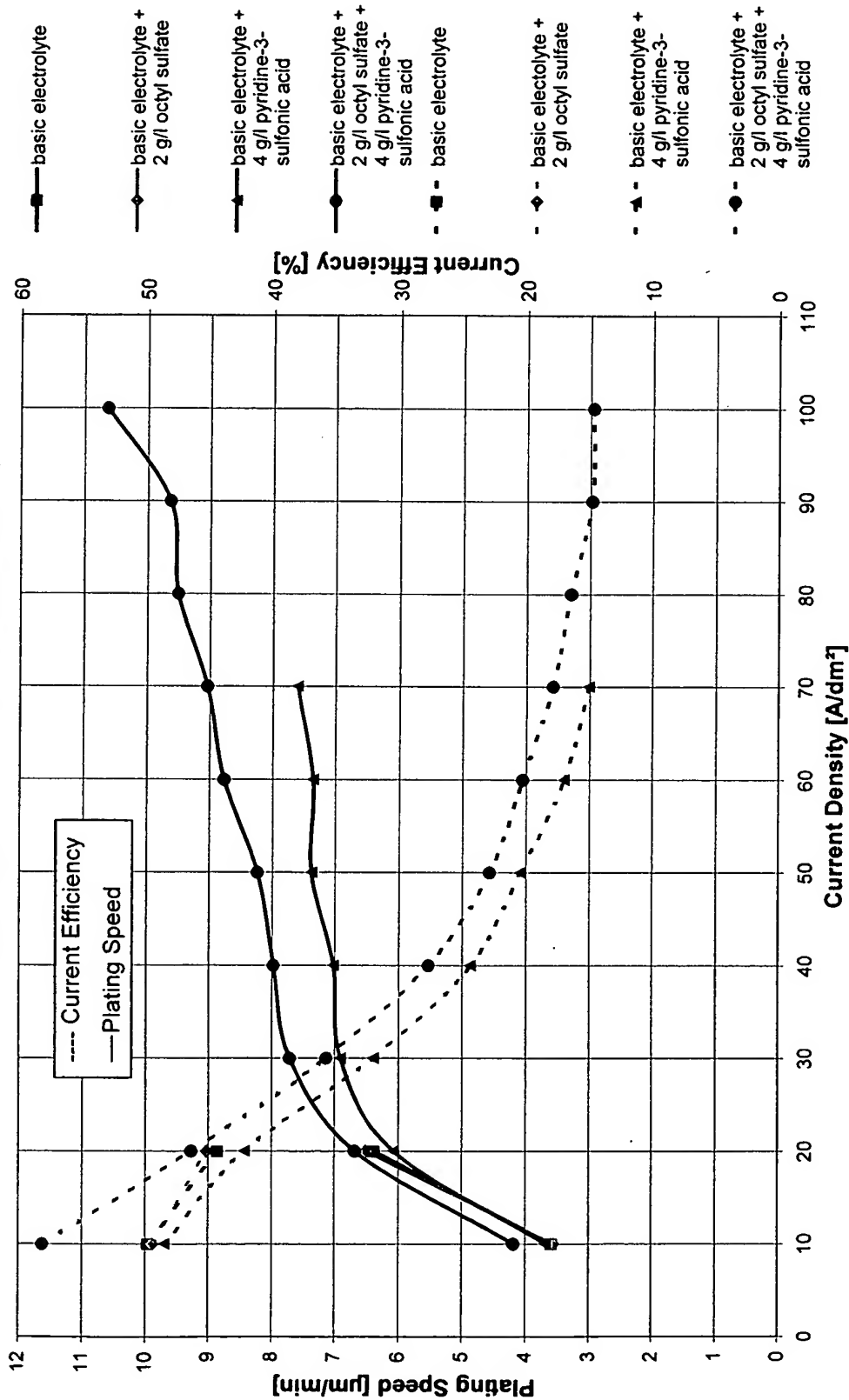


Figure 3

AuCo Electrolyte (12 g/l Au; 60 °C; pH 4.2; JetLab 600 I/h)

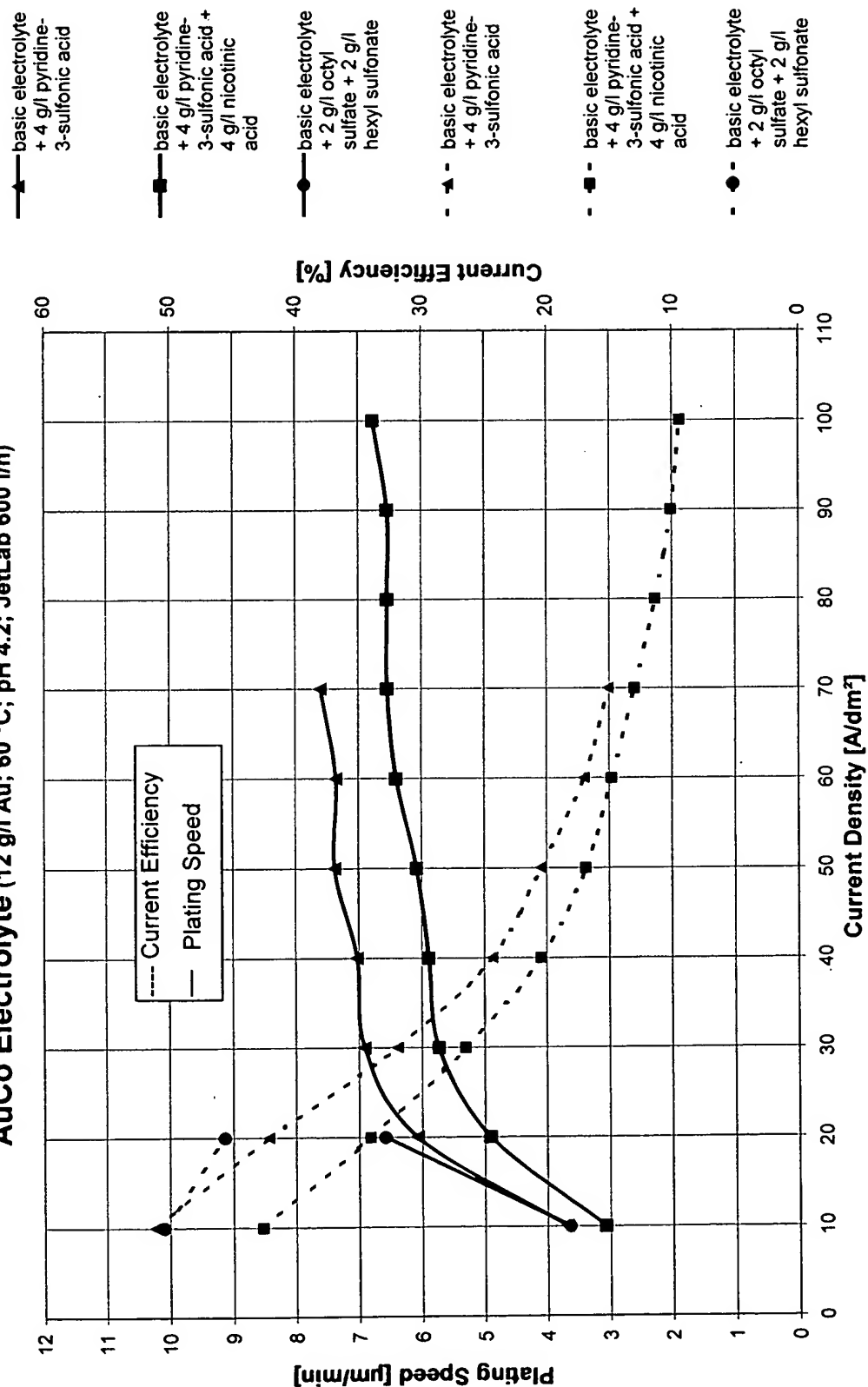


Figure 4